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CONTENTS

Uncertainty in the calibration of a partially immersed Liquid-in-Glass thermometer

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This paper presents an example of a typical calibration of a partially immersed Liquid-in-Glass (LiG) thermometer, by comparison to a standard precision digital thermometer, to obtain a correction to the LiG thermometer at a nominal temperature of 50° C. The standard way of evaluating and expressing the measurement uncertainty in typical corrections and uncertainty sources for the calibration has been used.

KEYWORDS: Calibration, Liquid-in Glass, thermometer.

Introduction

Calibration can be defined as a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realised by standards. Alternatively one can say that calibration is a comparison between a measuring standard, measuring instrument or equipment against a measuring standard of higher accuracy, to determine and document the value and accuracy of the quantity being compared.

A measurement standard is an object or instrument of which the relevant property has a known value (not to be confused with a *written* standard document like ISO 9000). The value of the standard has to be known to higher accuracy than the accuracy of the measuring instrument in order to be able to properly calibrate the instrument. For the value of the standard to be known, it also has to be determined (calibrated) by comparing it with higher level standards, which are calibrated and verified by comparison against still higher standards etc, up to the highest level, the international measurement standards. By this chain of comparisons from the measuring instrument all the way up to the highest world standards, the readings of all measuring instruments in the world will be equivalent and comparable to each other.

Since the result of a measurement is an approximation of the value of the quantity, that is the subject of the measurement (measurand), a complete declaration of the result must be accompanied by a statement of the quality of the measurement. Science and Technology presentations employ statements which can be quantified and verified. The uncertainty of measurement, evaluated according to the rules of the *Guide to the expression of uncertainty in measurement* (GUM) (Bureau International

des Poids et Mesures, 1995), has become a generally accepted quantitative measure of the accuracy of measurement results. The GUM defines the uncertainty of measurement as a parameter, associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand. The guide further makes it a pre-requisite for the technical equivalence of scientific laboratories that the uncertainty of measurement is evaluated and expressed in a standard way.

However, the GUM is by far, not a simple document, justifiably so because the issue of measurement uncertainty is a complex one. In fact there is a danger that some approaches can be so complex as to render them meaningful only to highly specialised metrology experts and statisticians who devise them, leaving the common industrial user confused. As such many metrologists (European Accreditation, 1997; National Measurement Accreditation Service (UK), 1995; Taylor and Kuyatt, 1994) are using alternatives to the document. Attempts to simplify the document have received considerable attention (Phillips and Eberhardt, 1997; Instone, 1996; Instone, 1993; Bell, 1996).

One way to facilitate the understanding of the GUM document is to outline its requirements through practical examples. This paper presents a complete, step by step, simplified practical procedure for the calibration of a partially immersed LiG thermometer, yet adhering to the strict requirements of the GUM in evaluating and expressing the uncertainty of measurement. Some typical uncertainty sources in temperature metrology have been considered in our evaluation of the combined uncertainty. It is hoped that this work will help encourage scientists and technicians to use the harmonised way of the GUM when evaluating and expressing the uncertainty of measurement.

Materials and Methods

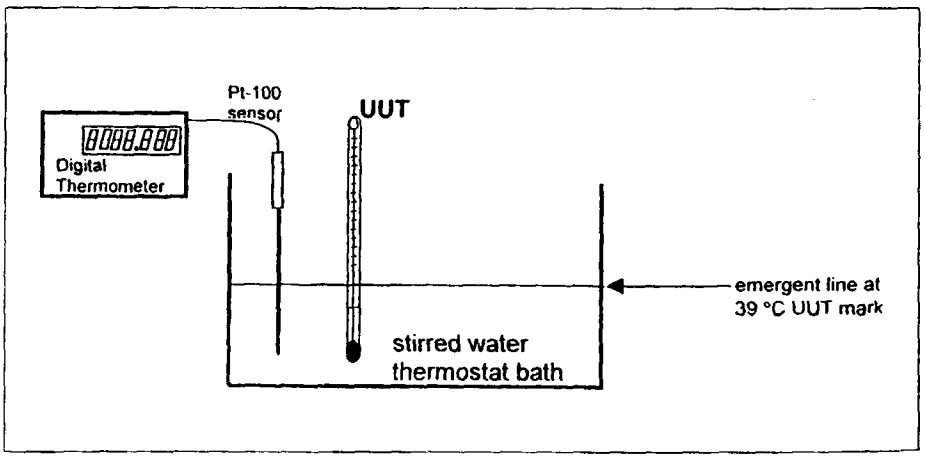

Figure 1: Experimental set-up for calibration of the LiG thermometer (UUT).

Under normal use, the LiG thermometer is immersed to the 39° C mark, and can measure to a maximum of 75° C. The smallest scale readings are 0,1° C.

The standard thermometer used comprised a digital readout unit and Standard Platinum Resistance Thermometer, calibrated as a system at several fixed points of the International Temperature Scale of 1990 (ITS-90). The standard has a normal operational range of –80° C to +400° C and the readout has a resolution of 0,001° C.

The water thermostat bath was stirred to maintain a nominal temperature of 50° C.

Procedure

The stability and homogeneity of the bath was determined by measuring the variation of temperature with time, and at several positions in the bath. Four sets, each of two readings (one from the standard and one from the LiG thermometer, the Unit Under Test (UUT)), were taken at the nominal temperature of 50° C. The temperature of the emergent liquid column of the LiG was estimated and the stem correction of the LiG calculated. The value of the correction for the UUT was then calculated. The method of the GUM was followed in evaluating all the measurement uncertainties.

Derivation of model equation

The temperature of the UUT is calculated using the following formula:

$$T_U = T_{U,R} + \Delta T_{RES} + \Delta T_U + C_S \qquad (1)$$

T_U = result.
$T_{U,R}$ = temperature reading on the UUT.
ΔT_{RES} = correction for the reading resolution (included to facilitate evaluation if its uncertainty contribution and has an assigned value of 0° C).
ΔT_U = calibration correction of the thermometer (at a nominal temperature of 50° C), the result of the calibration procedure described in this paper.
C_S = stem correction of the thermometer for the actual immersion depth.

The temperature, when measured with the digital thermometer is calculated by:

$$T_S = T_{S,R} + \Delta T_S \qquad (2)$$

T_S = result.
$T_{S,R}$ = temperature reading from the digital thermometer.
ΔT_S = calibration correction of the digital thermometer.

(The resolution of the readout is 0,001° C, which is negligible, so it is left out of the equation).

Under calibration the ΔT_U is determined by comparing the outcome of expression (1) with the known temperature from the (standard) digital thermometer by expression (2). However, there can be a difference between the temperatures T_U and T_S due to fluctuations and gradients in the thermostat bath:

$$T_U = T_S + \Delta T_B \qquad (3)$$

ΔT_B is the temperature difference due to the gradients and fluctuations in the bath. Substituting (1) and (2) into (3) gives:

$$T_{U,R} + \Delta T_{RES} + \Delta T_U + C_S = T_{S,R} + \Delta T_S + \Delta T_B \quad (4)$$

The correction of the UUT, to be determined by this calibration is therefore:

$$\Delta T_U = T_{S,R} + \Delta T_S + \Delta T_B - T_{U,R} - \Delta T_{RES} - C_S = (T_{S,R} - T_{U,R}) + \Delta T_S - \Delta T_{RES} - C_S + \Delta T_B \quad (5)$$

Four sets of readings are taken and the difference of each set ($T_{S,R}$ - $T_{U,R}$) calculated. The average of these four differences is combined with the corrections according to equation (5) to give the calibration correction ΔT_U of the UUT.

Evaluation of corrections and uncertainty sources

There are two different methods of evaluating the magnitude of uncertainty components. Type A evaluation consists of statistical analysis of the values obtained from several independent observations. In this paper, the type-A component is the experimental standard deviation of the average of the differences ($T_{S,R}$ - $T_{U,R}$), calculated from the readings (see Table 1).

Type B evaluation of the standard uncertainty is the method of evaluating the standard uncertainty by means other than statistical analysis of a series of observations. The evaluation in based on some other scientific knowledge.

The following type B uncertainty sources have been identified:

- Calibration uncertainty of the standard (from calibration certificate): $u(\Delta T_S)$.
- Resolution of the UUT: $u(\Delta T_{RES})$.
- Stem correction for the UUT (a function of the temperature): $u(C_S)$.
- Bath stability and homogeneity: $u(\Delta T_B)$.

Probability distributions

In order to be able to combine all uncertainty contributions they shall be expressed in the same form. The form chosen for this is the standard uncertainty, equivalent with the standard deviation from the type-A analysis.

The type of uncertainty source and the type of information available about it determine the way to calculate the standard uncertainty. The first step is to assume the most suitable distribution type, associated with the uncertainty source under consideration. A probability distribution shows the way in which the results are spread over the total range of values. The **normal** or Gaussian distribution is found when fluctuations of the values are due to a combination of many individual fluctuations, such as thermodynamic energy fluctuations. It is the most commonly found distribution. The highest probability is in the middle (the average). The range of possible values stretches to infinity, symmetrically on both sides, with the probability falling rapidly to very low values, the further we get away from the average (A). If we want to know the probability of finding a measurement value within a certain interval around the average we can take the surface area under the

graph over that interval. The parameter that is used as a reference is the standard deviation, indicated by the symbol s (sigma).

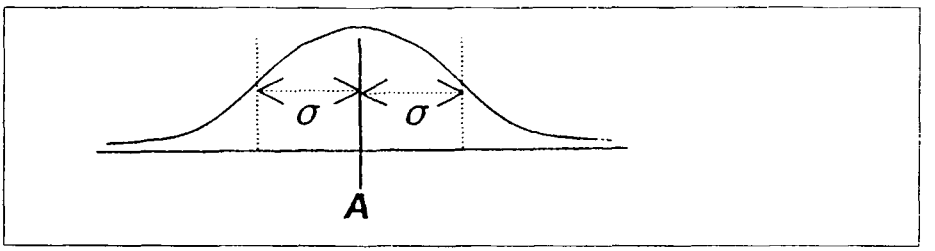

Figure 2: Normal distribution.

The mean of several readings follows a normal distribution, and is already at the one standard uncertainty level and was used in Table 2 to determine the type A uncertainty contribution.

The **uniform or rectangular** distribution occurs when there are clear boundaries beyond which the value cannot exist. The chance of the actual value being anywhere within the given interval is the same throughout the interval and zero outside the interval. The uniform distribution is often *assumed* if we do not know anything about the real distribution apart from some given "worst case" limits.

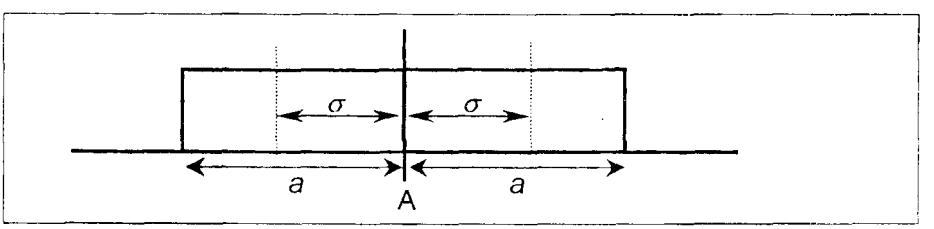

Figure 3: Rectangular or uniform distribution.

The formula for the standard deviation for a uniform distribution has been derived statistically by previous workers (Bureau International des Poids et Mesures, 1995; European Accreditation, 1997) :

$$\sigma = a/\sqrt{3} \qquad (6)$$

where σ = standard uncertainty

a = half-width or semi-range of the limits of the uncertainty component.

It is generally acceptable to treat the value of the uncertainty due to the resolution as if it is equally probable to lie anywhere within clear boundaries,

beyond which the value cannot exist, and thus model it by a uniform or rectangular probability distribution. In this paper we also assume a uniform distribution when evaluating the uncertainty contributions due to stem correction for the UUT and due to the bath stability.

Calibration correction and uncertainty of the standard
The calibration certificate of the digital thermometer states a correction at 50° C of - 0,010° C. The standard uncertainty, based on the calibration uncertainty from the certificate and an estimate of the drift since the date of calibration, is 0,010° C.

Thus ΔT_S = -0,010° C and $u(\Delta T_S)$ = 0,010° C

Resolution of the UUT
The scale of the LiG thermometer has 0,1° C divisions and can be interpolated to about 0,02° C. The estimate of the correction due to resolution is 0,000° C
The best estimate of the uncertainty is 0,5 × 0,02 = 0,010° C (half-width).
The standard uncertainty of a uniform distribution with a half-width of 0,010° C is calculated from equation (6).

Therefore $u(\Delta T_{RES})$ = 0,010 / √3 = 0,0058° C

Stem correction for the UUT
Since partial immersion mode was used, inevitably a length of mercury column protruded above the surface of the bath. This emergent liquid column (ELC) stands in temperature gradient whose mean temperature is variable and dependent on the ambient temperature. However, in the absence of auxiliary thermometers to measure the ELC temperature (t_{ELC}), an estimate of the temperature can be made.

Because the liquid column was very long, its average temperature was expected to be between the room temperature (t_{ROOM} = 28° C) and the temperature of the bath 50° C, such that 28° C ≤ t_{ELC} ≤ 50° C.

It was assumed to be likely that t_{ELC} was closer to the room temperature than the temperature of the bath. It was assumed therefore that t_{ELC} = 35 ± 5°C (Note the large uncertainty estimate here).

The stem correction to a thermometer's indication resulting from changes in ELC temperature is deduced from the relationship

$$C_S = K N (t_1 - t_2) \qquad (6)$$

- K is the apparent coefficient of expansion of mercury in glass (1,58 x 10^{-4} °C^{-1}).
- N is the length of the ELC expressed as a temperature in terms of the scale graduated on the individual thermometer or N is the difference between the measured temperature and the temperature reading at the immersion line (i.e. N = 50° C - 39° C = 11° C).
- t_1 is the temperature of the ELC as specified on the calibration certificate (20° C).
- t_2 is the measured temperature of the ELC (estimated to be 35° C in this example).

Therefore:
$t_1 - t_2 = 20° C - 35° C = -15° C.$

Hence the stem correction becomes
$C_s = 1,58 \times 10^{-4°} C^{-1} \times 11°C \times (-15° C) = -0,026° C.$

Calculation of uncertainty in stem correction
N has negligible uncertainty, compared with the other contributions.
The uncertainty of $t_1 - t_2$ is equal to the uncertainty of t_2 because the uncertainty in t_1 is negligible.
Hence $u(t_1 - t_2) = u(t_2) = 5° C.$
and $u(C_s) = k \times N \times u(t_2) = 1,58 \times 10^{-4°} C^{-1} \times 11° C \times 5° C = 0,009° C.$
Assuming a uniform or rectangular distribution, we have: $u(C_s) = 0,009 / \sqrt{3} = 0,005° C.$

Bath homogeneity and stability
The standard thermometer was used to investigate the bath homogeneity by measuring the temperature at different positions in the bath. The standard could not resolve any temperature gradients, which were assumed to be less than the resolution of the standard thermometer and hence negligible.

To determine the uncertainty contribution from the bath stability, readings from the digital thermometer were recorded for several cycles of the bath temperature.
The following graph shows an approximation of the variation of temperature with time observed while monitoring the bath stability. The temperature was measured by the standard thermometer.

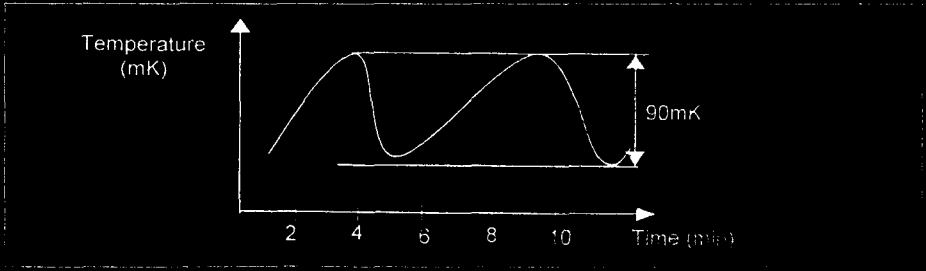

Figure 4: Variation of bath temperature with time.

It was not possible to determine the sign of the correction [illegible], so the estimated value was 0° C.

A uniform distribution was assumed, since the temperature [illegible] within the fixed limit of 90 mK = 0,09° C.
The uncertainty contribution from t_b is calculated from [illegible]

$u(t_b) = 0,098° C / 2 = 0,045° C.$

This gives the standard uncertainty due to the bath stability as:

$u(DT_B) = 0{,}045°\ C/\sqrt{3} = 0{,}026°\ C$ for an assumed uniform distribution.

Sensitivity coefficients

Sensitivity coefficients are used to quantify the influence that each of the uncertainty components has on the final result, i.e. how sensitive the result is to each of the influence quantities. In this example all the terms in equation (5) are in ° C, and the result is simply the sum of these terms, so all sensitivity coefficients are equal to 1.

Degrees of freedom

The degrees of freedom is a parameter that gives a measure of how reliably the standard uncertainty has been estimated and is used to determine the coverage factor that corresponds to a specified coverage probability.

The effective degrees of freedom ν_{eff} in the combined uncertainty can be determined by using the Welch-Satterthwaite equation (Bureau International des Poids et Mesures, 1995; European Accreditation, 1997)

$$\nu_{eff} = \frac{(u_c(\Delta T_U))^4}{\sum_{i=1}^{N} \frac{(u_i(\Delta T_U))^4}{\nu_i}} = \frac{(u_c(\Delta T_U))^4}{\frac{(u_1(\Delta T_U))^4}{\nu_1} + \frac{(u_2(\Delta T_U))^4}{\nu_2} + \ldots + \frac{(u_N(\Delta T_U))^4}{\nu_N}} \qquad (7)$$

$u_c(\Delta T_U)$ is the combined standard uncertainty in the correction, obtained from all the uncertainty contributions by the root sum square method (calculated in Table 1 to be 0,035° C).

ν_i represents the effective degrees of freedom of the standard uncertainty contribution $u_i(\Delta T_U)$ with $i = 1$ to N.

In general the number of degrees of freedom for type B components (with uniform or rectangular distributions) can be considered to be infinite, the assumption being that we have chosen our limits such that the probability of the quantity in question lying outside limits is extremely small.

All uncertainty contributions with infinite degrees of freedom fall out of the sum (equation (7)) because these terms become zero (division by infinity), except for the type-A component, which has $\nu_i = n\text{-}1 = 3$ degrees of freedom, and is inevitably dependent on n, the number of observations.

The effective degrees of freedom therefore simplify to:

$$\nu_{eff} = u_c(\Delta T_U)^4/(u(\Delta T_U)^4/3) = 0.035^4/(0.020^4/3) = 28$$

after rounding the outcome of the formula down to the nearest lower integer number. On looking up the nearest value (25) in Table 3 of Student's-t factors, it is observed that the coverage factor for 95 percent confidence level is 2.06.

The expanded uncertainty is simply the total uncertainty expressed as a value with a given confidence level and is obtained by multiplying the combined standard uncertainty by the coverage factor.

Therefore expanded uncertainty (95 percent confidence level) = 0.035° C x 2.06 = 0.072° C.

The uncertainty analysis or budget for the calibration is given in Table 1 below, and includes a list of all the sources of uncertainty together with the associated standard uncertainties and the methods used to evaluate them. The table summarises all the calculations involved in finding the correction to the UUT, its standard uncertainty and the effective degrees of freedom.

Table 1: The uncertainty analysis/budget as required by the GUM.

Quantity X_i	Estimate x_i	Uncertainty $u(x_i)$	Distribution	Standard uncertainty $u(x_i)$	Sensitivity coefficient C_i	Uncertainty contribution $u_i(y)$	Degrees of freedom n_i
ΔT_S	- 0,010 °C	0,020° C	normal	0,010° C	1	0,010° C	•
ΔT_{RES}	0,000 °C	0,010° C	uniform	0,006° C	1	0,006° C	•
C_S	- 0,026 °C	0,009° C	uniform	0,005° C	1	0,005° C	•
ΔT_B	0,000 °C	0,045° C	uniform	0,026° C	1	0,026° C	•
$(T_{S,R} - T_{U,R})$	0.055° C	type-A	normal	0,020° C	1	0,020° C	3
Result							
Y	y					$u_c(y)$	ν_{eff}
ΔT_U	0,019° C	Combined standard uncertainty fi				0,035° C	28

Results

Reported result of calibration

At temperature T = 50° C, the correction for the LiG thermometer is

$$\Delta T_U = 0.02 \pm 0.07° \text{ C}.$$

The indicated uncertainty has confidence level of 95 percent, for an assumed normal distribution with effective degrees of freedom of 28.

Table 2: Measured readings of the standard and UUT and the calculated difference, and standard deviations.

Nominal Pemp	Reading NO	Reading Standard $T_{S,R}$	Reading UUT $T_{U,R}$	Difference $(T_{S,R} - T_{U,R})$
50	1	50.06	49.95	0.110
50	2	50.02	50.00	0.020
50	3	50.07	50.01	0.060
50	4	50.03	50.00	0.030
SUM				0.220
MEAN				0.055
			Standard Deviation	0.040
			Standard deviation of mean	0.020

Discussion

Table 3: Value of t_p (ν) from the *t*-distribution for degrees of freedom ν that defines an interval $-t_p$ (ν) to $+t_p$ (ν) that encompasses the fraction *p* of the distribution.

Degrees of freedom	Fraction *p* in percent					
ν	68.27[a]	90	95	95.45[a]	99	99.73[a]
1	1.84	6.31	12.71	13.97	63.66	235.80
2	1.32	2.92	4.30	4.53	9.92	19.21
3	1.20	2.35	3.18	3.31	5.84	9.22
4	1.14	2.13	2.78	2.87	4.60	6.62
5	1.11	2.02	2.57	2.65	4.03	5.51
6	1.09	1.94	2.45	2.52	3.71	4.90
7	1.08	1.89	2.36	2.43	3.50	4.53
8	1.07	1.86	2.31	2.37	3.36	4.28
9	1.06	1.83	2.26	2.32	3.25	4.09
10	1.05	1.81	2.23	2.28	3.17	3.96
11	1.05	1.80	2.20	2.25	3.11	3.85
12	1.04	1.78	2.18	2.23	3.05	3.76
13	1.04	1.77	2.16	2.21	3.01	3.69
14	1.04	1.76	2.14	2.20	2.98	3.64
15	1.03	1.75	2.13	2.18	2.95	3.59
16	1.03	1.75	2.12	2.17	2.92	3.54
17	1.03	1.74	2.11	2.16	2.90	3.51
18	1.03	1.73	2.10	2.15	2.88	3.48
19	1.03	1.73	2.09	2.14	2.86	3.45
20	1.03	1.72	2.09	2.13	2.85	3.42
25	1.02	1.71	2.06	2.11	2.79	3.33
30	1.02	1.70	2.04	2.09	2.75	3.27
35	1.01	1.70	2.03	2.07	2.72	3.23
40	1.01	1.68	2.02	2.06	2.70	3.20
45	1.01	1.68	2.01	2.06	2.69	3.18
50	1.01	1.68	2.01	2.05	2.68	3.16
100	1.005	1.660	1.984	2.025	2.626	3.077
∞	1.000	1.645	1.960	2.000	2.576	3.000

[a] For a quantity *z* described by a normal distribution with expectation μ_z and standard deviation s, the interval $\mu_z \pm k\sigma$ encompasses p = 68.27, 95.45, and 99.73 percent of the distribution for k = 1, 2, and 3, respectively.

The outlined procedure can be generalised and used for the evaluation of uncertainty measurements in science and industry where quality is of importance. Tolerances are becoming ever tighter and more accurate measurements needed. More

sophisticated measuring equipment is needed. Conformance to specifications has become a major requirement. The Standard approach of the GUM is important to avoid confusions which result from use of non-standard methods to quantify the quality of a measurement.

The report of the result of a calibration process, including the uncertainty statement, must give all information needed for interpretation, namely, uncertainty value, distribution type, confidence level or coverage factor, and (sometimes) degrees of freedom.

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